
कार्बन टेट्राक्लोराइड — विशिष्टि
(तीसरा पुनरीक्षण)

**Carbon Tetrachloride —
Specification**
(*Third Revision*)

ICS 71.080.20

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FOREWORD

This Indian (Third Revision) Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Carbon Tetrachloride is used extensively as a solvent and diluent. It is also used for the manufacture of refrigerants of chlorofluoromethane series, in grain fumigants, in fire extinguishing blends and for moth control purposes.

This standard was first published in 1955 and subsequently revised in 1970 and 1977. The first revision was taken up to formulate two grades of the material and requirement for methyl chloride, methylene chloride and chloroform content were included.

In the second revision, the requirements for relative density and distillation range for both the grades were modified. For Grade 1 the non-volatile matter content was changed. The characteristic of colour was included. Further, test method for determination of Sulphur compound modified to spectrophotometer method.

In this (*third revision*), Grade 2 for technical grade has been deleted, as it is not manufactured nowadays. The major changes in this revision are as under:

- a) The characteristic of purity, which is tested by gas chromatography has been incorporated;
- b) Gas chromatography test method has been modified for impurity and purity for better separation, low level detection and quantification of impurities, as gas chromatography method is more authentic and accurate as compared to distillation method;
- c) Free chlorine and acidity or alkalinity methods have been modified and alternative test method for color determination has been incorporated; and
- d) With the advancement in technologies in production of carbon tetrachloride, carbon disulfide (CS₂) is not produced nowadays and hence carbon disulphide characteristics has been deleted from the specifications.

The composition of the committee, responsible for the formulation of this standard is listed in Annex K.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CARBON TETRACHLORIDE — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for carbon tetrachloride (CCl₄).

2 REFERENCES

The standards listed below, contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (<i>second revision</i>)
IS 2837 (Part 2) : 1977	Specification for porcelain crucibles and basins: Part 2 Basins (<i>first revision</i>)
IS 4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)
IS 5311 : 1969	Code of safety for carbon tetrachloride
IS 8768 : 2000	Method of measurement of colour in liquid chemical products platinum — Cobalt scale (<i>second revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall consist essentially of carbon tetrachloride (CCl₄) and shall be a clear liquid free from sediment and suspended matter.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col (4) and col (5) of Table 1.

3.2.1 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 PRECAUTIONS IN HANDLING

Widespread application of carbon tetrachloride often without evidence of ill-effects has resulted in underrating its toxicity. Careful investigations have shown carbon tetrachloride to be one of the most harmful of the common solvents. Extreme care, therefore, shall be taken in handling it (*see* 5.2.2 and IS 5311).

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed as agreed to between the purchase and the supplier. It is recommended that, as a finished product, carbon tetrachloride, be handled in galvanized or epoxy-lined mild steel containers or HM-HDPE drums rather than plain steel and cast-iron drums.

5.1.2 The material may be also transported in mild steel or stainless-steel road tanker which shall be free from contaminations like oil and grease, rust, etc. Main holes and loading and unloading points shall be hermetically sealed with suitable non-reacting gaskets like PTFE and NAF etc.

5.1.3 The material shall be stored in original container, protected from direct sunlight. Containers shall be tightly closed and sealed until ready for use. Containers that have been opened must be carefully resealed and kept upright to prevent leakage.

5.2 Marking

5.2.1 Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of manufacturer and his recognized trade-mark, if any;

- c) Month and year of manufacture;
- d) Lot or batch number;
- e) Net weight; and
- f) Any other statutory requirements.

5.2.2 All containers in which the material is stored or transported shall be suitably marked with word 'TOXIC'. The label shall also be prominently and clearly marked with the following marking:

**STORE IN COOL AND DRY PLACE.
PROTECT FROM DIRECT SUNLIGHT.**

**USE WITH ADEQUATE VENTILATION,
WEAR MASK AND NECESSARY
PERSONAL PROTECTIVE EQUIPMENTS
DURING HANDLING**

5.2.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may also be marked with the Standard Mark.

6 SAMPLING

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex J.

Table 1 Requirements for Carbon Tetrachloride
(Clause 3.2)

Sl No.	Characteristics	Requirements	Method of test, Ref to	
			Annex (4)	IS No. (5)
(1)	(2)	(3)		
i)	Relative density at 27 °C/27 °C	1.584 - 1.590	A	—
ii)	Colour, Pt-Co, <i>Max</i>	15	—	IS 8768
iii)	Distillation range at 760 mmHg ¹⁾	2 to 97 ml shall distil between 76.0 to 77.0 °C	B	—
iv)	Non-volatile matter, percent by mass, <i>Max</i>	0.002	C	—
v)	Purity, percent by mass, <i>Min</i>	99.5	D	—
vi)	Methyl chloride, Methylene chloride and Chloroform, percent by mass, <i>Max</i>	0.025	D	—
vii)	Moisture content, percent by mass, <i>Max</i>	0.01	E	—
viii)	Acidity (<i>as</i> HCl) or alkalinity (<i>as</i> NaOH), ppm, <i>Max</i>	5.0	F	—
ix)	Spot test	To pass test	G	—
x)	Free Chlorine	To pass test	H	—

¹⁾ Distillation range at 760 mmHg is an optional parameter or as agreed between the purchaser and the manufacturer.

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF RELATIVE DENSITY

A-1 OUTLINE OF THE METHOD

In this method, mass of equal volumes of the material and water at the same temperatures are compared using relative density bottle.

A-2 APPARATUS

A-2.1 Relative Density Bottle, with well-fitting ground-glass joints.

To calibrate, clean and dry the bottle thoroughly, weigh and then fill with water, freshly boiled and cooled to 27 °C, after removing the cap. Fill to overflowing by holding the bottle on its side in such a manner as to prevent entrapment of air bubbles, insert the stopper and immerse in a water-bath at (27.0 ± 0.2) °C. Keep the entire bulb completely covered with water and hold at that temperature for 30 min.

Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to a lower temperature and weigh. Calculate the mass of water. The relative density bottle of about 50 ml capacity and of either of the two shapes, as shown in Fig. 1 is recommended.

A-2.2 Water Bath, maintained at (27.0 ± 0.2) °C.

A-2.3 Thermometer, any convenient thermometer of a suitable range with 0.1 °C or 0.2 °C subdivisions.

NOTE — The thermometer shall bear a certificate from any institution authorized to issue certificate traceable to international or national measurement standards.

A-3 PROCEDURE

Fill the relative density bottle with the material previously kept at about 27 °C to overflowing, holding the bottle on its side in such a manner as to prevent entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water-bath maintained at (27.0 ± 0.2) °C and hold for 30 min. Carefully wipe off any liquid which has come through the capillary opening. Remove the relative density bottle from the bath, clean dry it thoroughly, cool and weigh.

A-4 CALCULATION

$$\text{Relative density at } 27^\circ\text{C}/27^\circ\text{C} = \frac{A-B}{C-B}$$

where

A = mass of the relative density bottle filled with the material at 27 °C, in g;

B = mass of the relative density bottle, in g; and

C = mass of the relative density bottle filled with water at 27 °C, in g.

NOTES

1 The relative density determined at any temperature within the range 25 °C to 35 °C can be adjusted to 27 °C/27 °C using the correction factor of + 0.0 016 for every degree celsius fall, and - 0.0 016 for every degree celsius rise in temperature.

2 Digital density meter may also be used.

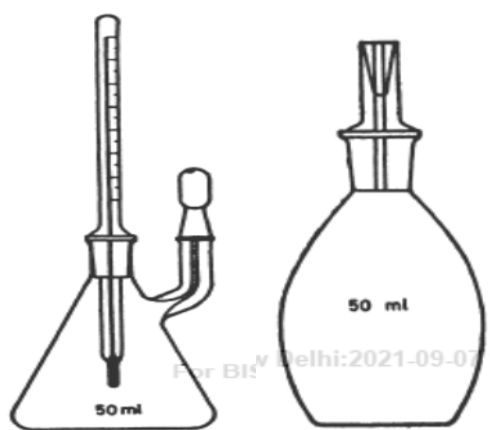


FIG. 1 RELATIVE DENSITY BOTTLE

ANNEX B

[Table 1, Sl No. (iii)]

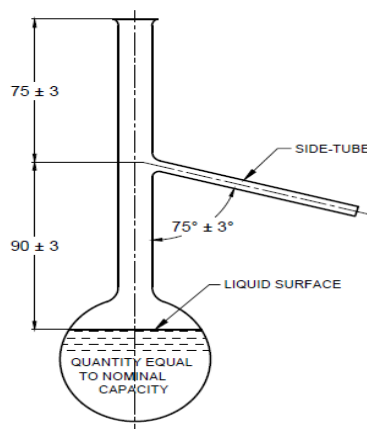
DETERMINATION OF DISTILLATION RANGE

B-1 OUTLINE OF THE METHOD

The material is distilled in the specified apparatus under the prescribed condition of heat input and rate of distillation of 100 ml of the material distilled and percentages by volume of the distillate recovered at the specified temperatures are recorded.

B-2 APPARATUS

B-2.1 Distillation Flask, of the shape and dimensions shown in Fig. 2.



All dimensions in millimeters
FIG. 2 DISTILLATION FLASK

B-2.2 Thermometer, a suitable partial immersion thermometer having a range of 48 °C to 100 °C, graduated at each 0.2 °C and with a maximum scale error of 0.2 °C. The maximum overall length of the thermometer shall be 385 mm and stem diameter shall be 5.5 mm to 8.0 mm.

NOTE — The thermometer shall bear a certificate from any institution authorized to issue certificate traceable to international or national measurement standards.

B-2.3 Rectangular Draught Screen, rectangular in cross-section, made of 0.8 mm thick sheet metal, with dimensions as shown in Fig. 3 and open at the top and bottom. It shall comply with the following requirements:

- a) In each of the two narrower sides of the draught screen there shall be two circular holes, each 25 mm diameter, and in each of the four

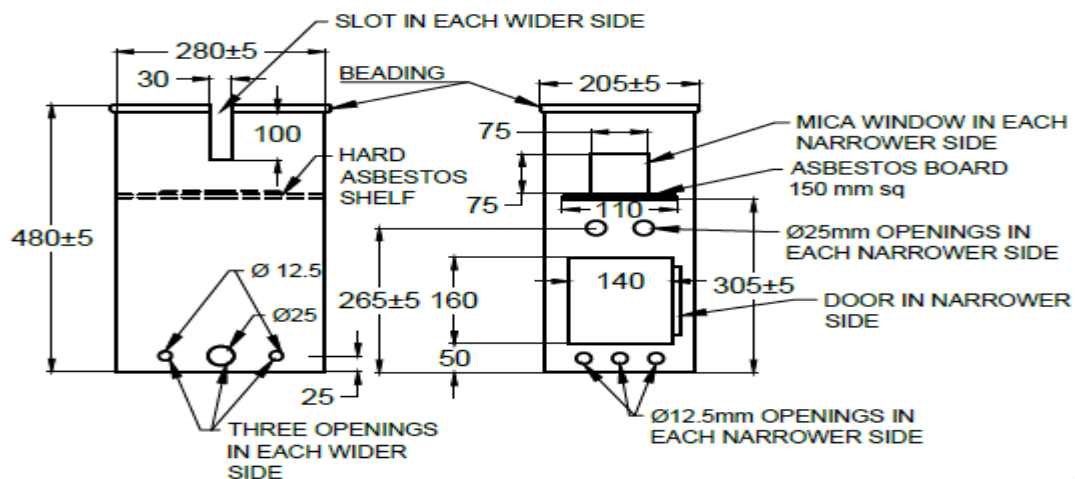
sides of the draught screen there shall be three holes with their centers 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 3. The diameter of each of the holes centrally situated in the longer sides shall be 25 mm and of the remaining ten holes shall be 12.5 mm. At the middle of each of the wider sides a vertical slot with the dimensions as shown in Fig. 3 shall be cut downwards from the top of the screen. A removable shutter conforming to the dimensions in Fig. 4 shall be provided for closing the vertical slot not in use.

- b) A shelf of hard asbestos board, 6 mm in thickness and having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

- c) In one of the narrower sides of the screen a door shall be provided having dimensions and position as shown in Fig. 3. In each of the narrower sides of the screen a mica window shall be placed centrally with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 4.

- d) An asbestos board 150 mm × 150 mm × 6 mm in size having a central hole 50 mm in diameter shall be so placed on the asbestos shelf that the two holes are approximately concentric and the distillation flask when in position completely closes the hole of the asbestos board.

B-2.4 Liebig Condenser, with the bent end made and wall thickness of 1.0 mm to 1.5 mm and conforming to the shape and dimensions shown in Fig. 5. The bent portion may, however, be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.



All dimensions in millimeters
FIG. 3 RECTANGULAR DRAUGHT SCREEN

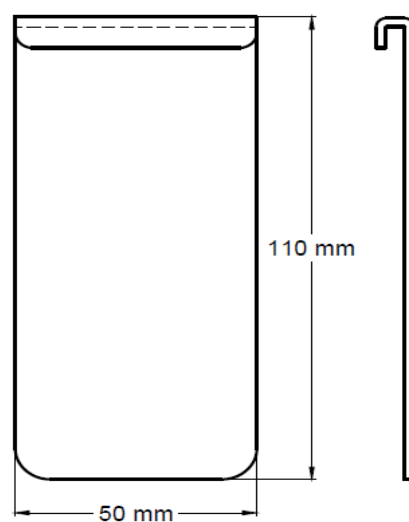
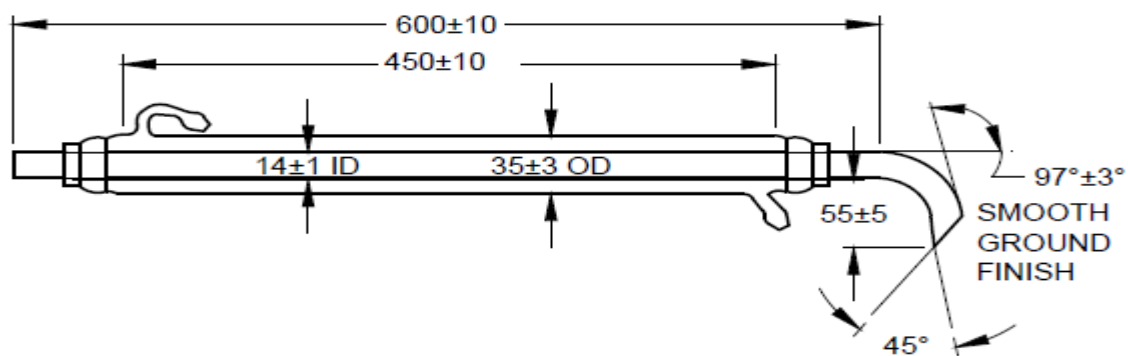
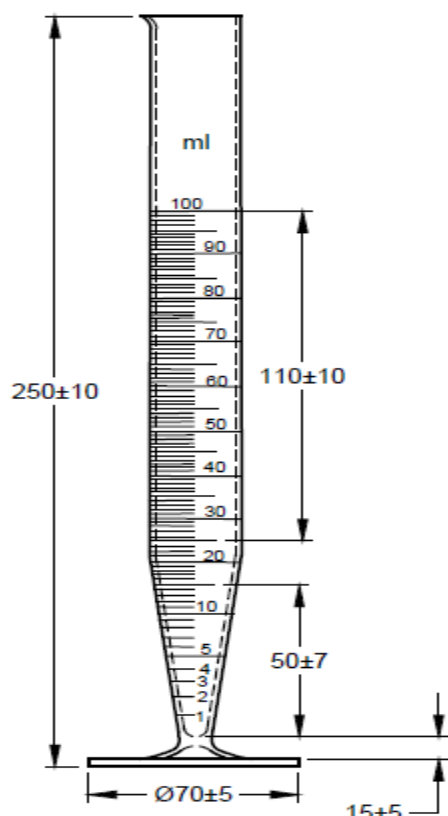


FIG. 4 REMOVABLE SHUTTER



All dimensions in millimeters
FIG. 5 LIEBIG CONDENSER

B-2.5 Receiver, of 100 ml capacity, with dimensions and graduation shown in Fig. 6.



All dimensions in millimeters
FIG. 6 RECEIVER

B-2.6 Electric Heater, Gas Burner or other Flame Type Heater, any suitable heater or burner that enables the distillation to be carried out as described in B-3.

B-3 PROCEDURE

Assemble the apparatus as shown in Fig. 7. Measure 100 ml of the material at laboratory temperature by means of the receiver and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Fit the thermometer in the

flask so that the bottom of the capillary is in level with the lower edge of the side-tube joint. Pass an adequate supply of cooling water at 0 °C to 5 °C, through the condenser. To receive the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly; especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be not less than 5 min nor greater than 10 min. Place the receiver in a manner that the condensate flows down its side. Continue the distillation at the rate of 4 ml/min to 5 ml/min (about 2 drops). Read the temperature on the thermometer when 2 ml and 97 ml of the distillate have been collected in the cylinder.

B-4 CORRECTION OF THE THERMOMETER READING

B-4.1 Error of Scale

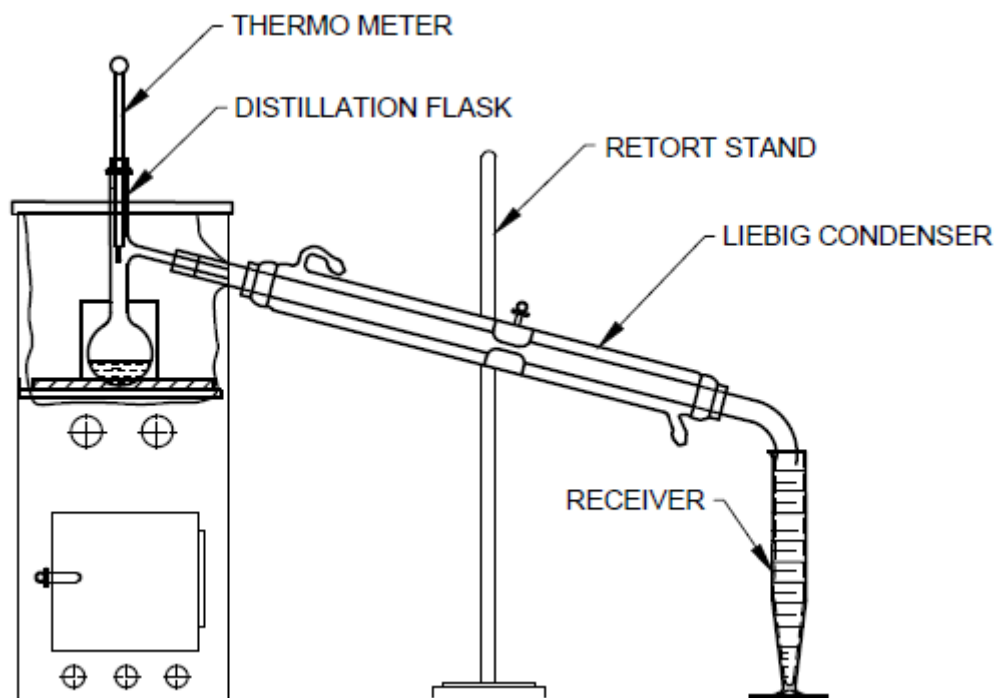
In all the thermometer readings, make the corrections as indicated on the certificate of the instrument.

B-4.2 Correction for Barometric Pressure

If the barometric pressure prevailing during the determination is normal, namely, 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected under B-4.1 shall be used as such. If, however, the prevailing pressure deviates from 760 mmHg, the specified temperatures shall also be corrected as follows:

- For every 10 mmHg above 760 mmHg, subtract 0.43 °C from the specified temperature; and
- For every 10 mmHg below 760 mmHg, add 0.43 °C to the specified temperature.

NOTE — These corrections are valid only for pressures above 700 mmHg.



All dimensions in millimeters

FIG. 7 ASSEMBLY OF APPARATUS FOR DETERMINING DISTILLATION RANGE

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF NON-VOLATILE MATTER

C-1 OUTLINE OF THE METHOD

A known quantity of the material is evaporated on a water-bath and the residue is weighed.

C-2 APPARATUS

C-2.1 Porcelain Basin, 150 ml capacity
[see IS 2837(Part 2)]

C-2.2 Water Bath

C-2.3 Oven, capable of maintaining temperature between 105 °C to 110 °C.

C-3 PROCEDURE

Clean the basin and dry in a desiccator. Weigh the empty basin (previously conditioned in the oven at 105 °C to 110 °C for 30 min and cooled in a desiccator) to the nearest 0.0 002 g (M_1).

Take 100 ml of the material in the basin. Evaporate the material over a water bath in a fume cupboard to almost dryness. Further heat in the oven at 105 °C to 110 °C for 30 min. Cool in a desiccator and weigh accurately (M_2). Repeat the heating for 15 min, followed by cooling and weighing till, two consecutive weighing do not differ by more than 0.5 mg.

C-4 CALCULATION

Non-volatile matter, percent by mass = $\frac{M}{v \times d} \times 100$
where

M = mass, of the residue obtained ($M_2 - M_1$), in g;

v = volume, of the material taken for the test, in ml; and

d = relative density, of the material (see Annex B).

ANNEX D

[Table 1, Sl No. (v) and (vi)]

DETERMINATION OF PURITY, METHYL CHLORIDE, METHYLENE CHLORIDE AND CHLOROFORM BY GAS CHROMATOGRAPHY

D-1 APPARATUS

D-1.1 Gas Chromatograph

D-1.1.1 Any gas chromatograph equipped with a

flame ionization detector (FID), a split-splitless injector and a suitable electronic integrator/software may be used with following accessories and operating condition:

Column	: Column of fused silica, with 6 percent cyanopropyl phenyl and 94 percent dimethylpolysiloxane with length: 30 m; internal diameter: 0.53 mm and film thickness: 3.0 μm
Carrier gas and flow rate (ml/min)	: Hydrogen (H_2) and 10 ml/min
Split ratio	: 10 : 1
Make up (N_2) flow rate ml/min	: 25
Hydrogen flow rate	: 30 ml/min
Zero air flow rate	: 300 ml/min
Septum purge flow rate	: 3 ml/min
Sample size	: 2 μl (may vary as per user method suitability)
Detector	: Flame ionization detector (FID)

D-1.1.2 Temperature Program of Oven, Detector and Injector:

Injector Temperature, $^{\circ}\text{C}$	Detector Temperature, $^{\circ}\text{C}$	Oven		
		Temperature, $^{\circ}\text{C}$	Hold Time, min	Ramp Rate, $^{\circ}\text{C}/\text{min}$
250 \pm 1	250 \pm 1	40 \pm 1	5	10
		160	1	—

NOTES

1 The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He , H_2 , N_2) may be used with applicable GC operating

parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

2 Helium or Hydrogen or Nitrogen may be used as the carrier gas. The operating parameters of the gas chromatograph given above are according to hydrogen, as carrier gas.

D-1.2 Syringe, 10 µl capacity

D-1.3 Recorder, chromatogram is displayed on the computer screen

D-2 REAGENTS**D-2.1 Acetone****D-2.2 Synthetic Standard of Pure Carbon Tetrachloride**

For synthetic standard, mix methyl chloride, methylene chloride and chloroform in pure carbon tetrachloride as given below:

<i>Component</i>	<i>Concentration, percent by mass</i>
Methyl chloride	0.008 – 0.009
Methylene chloride	0.008 – 0.009
Chloroform	0.008 – 0.009
Carbon tetrachloride	Balance

D-3 PROCEDURE**D-3.1 Sample Injection**

Rinse 10 µl syringe with acetone twice and dry for 2 min or longer. Rinse the syringe twice with the synthetic standard. Inject standard solution (as per sample size defined in **D-1.1.1**) into the gas chromatograph and obtain a standard chart. Rinse the syringe twice with the material under test and inject the same sample size used for standard solution into the gas chromatograph. It is imperative that the same injection technique is used, and the syringe used in the operation is not used with any other type of solvent. It is very necessary to prevent contamination.

D-3.2 The components will elute in the order of their boiling points:

- First — Methyl chloride;
- Second — Methylene chloride;
- Third — Chloroform; and
- Fourth — Carbon tetrachloride.

D-4 CALIBRATION

Internal normalization is the method used for calibration of the results of the analysis. Because of low contents of unknown impurities, it is not necessary to use correction factors for converting the percentage of the areas into percentage by mass, so that these factors in the calculation formula have the value $R_f = 1$. In case of examples with higher contents of known impurities, there is a need for determining correction factors; this should be done by using a synthetic standard solution. The mixture is run through the chromatograph. This would give the positions at which each component would elute. By comparison of the actual values obtained for the standard and the composition of the standard prepared the correction factor for the component may be calculated.

D-5 DETERMINATION OF RESPONSE FACTOR (R_f)

D-5.1 Inject synthetic standard mixture (**D-2.2**) three times, one by one and read the response/area percentage of methyl chloride, methylene chloride and chloroform. Calculate the response factor for impurities in standard by using given formula,

Response factor (R_f) of methyl chloride, methylene chloride and chloroform:

$$R_f = \frac{\text{Actual concentration (percent of interested impurity)}}{\text{Area percent from GC}}$$

D-5.2 Calculate the average response factor for all individual impurity as average of three readings. The final response factor of individual impurity shall be taken for the calculation of impurities concentration in chloroform.

D-6 CALCULATION**D-6.1 For Impurities**

Interested impurity, percent = (Area percent of Interested Impurity) x (R_f of Interested Impurity)

D-6.2 For Purity

Purity, percent = 100 – (Sum of all impurities)

ANNEX E

[Table 1, Sl No. (vii)]

DETERMINATION OF MOISTURE CONTENT

E-1 Take 100 ml of the material and determine its moisture content as prescribed in IS 2362.

NOTE — As an alternative test method, Karl Fischer (KF) auto titrator may also be used.

ANNEX F

[Table 1, Sl No. (viii)]

DETERMINATION OF ACIDITY (*as HCl*) OR ALKALINITY (*as NaOH*)

F-1 PREPARED AQUEOUS EXTRACT

Shake 25 ml of the material with 25 ml of freshly boiled and cooled water for 3 min and allow the layers to separate. Reserve the aqueous layer for the test.

F-2 REAGENTS

F-2.1 Methyl Orange Indicator

Dissolve 0.50 g of the methyl orange in water and dilute to 1 litre.

F-2.2 Bromothymol Blue Indicator Solution

Dissolve 50 mg of bromothymol blue in 4 ml of 0.02 M sodium hydroxide and 20 ml of ethanol

(95 percent). After the solution is effected, add sufficient water to produce 100 ml.

F-3 PROCEDURE

Take 10 ml of the prepared aqueous extract (**F-1**) and add 4 drops of methyl orange indicator. Carry out a control test by adding the same amount of indicator to 10 ml of freshly boiled and cooled distilled water and compare the colour produced in both. Repeat this test using 4 drops of bromothymol blue solution in place of methyl orange indicator.

F-4 The material shall be taken as neutral if the colour obtained with the prepared aqueous extract shows no difference when compared with the colour produced in the control test.

ANNEX G

[Table 1, Sl No. (ix)]

SPOT TEST

G-1 PROCEDURE

G-1.1 Place 5 drops of the material near the center of a filter paper (Whatman No. 40 or equivalent), taking care that the wet portion of the filter paper does not come into contact with any object. Allow

to evaporate in a well-ventilated room and keep for 2 h.

G-1.2 The material shall be taken to have satisfied the test if no spot or stain is left on the filter paper.

ANNEX H

[Table 1, Sl No. (vii)]

TEST FOR FREE CHLORINE

H-1 OUTLINE OF THE METHOD

The material is shaken with potassium iodide solution (H-3.1) and the blue colour developed, is noted.

H-2 APPARATUS

H-2.1 Graduated Measuring Cylinder, 25 ml capacity, glass stoppered (*see* IS 878)

H-3 REAGENT

H-3.1 Potassium Iodide Solution

Dissolve 100 g potassium iodide in 1 000 ml of

distilled water. Store the solution in the dark.

H-4 PROCEDURE

H-4.1 Place 5 ml of sample in a ground glass stopper tube. Add 5 ml of potassium iodide solution and 0.2 g of soluble starch. Shake the tube for 30 s and keep in the dark for 5 min.

NOTE — The test shall be carried out in the dark and colour developments shall be checked immediately.

H-4.2 The material shall be regarded to have passed the test if no blue colour is developed within 5 min.

ANNEX J

(Clause 6)

SAMPLING OF CARBON TETRACHLORIDE

J-1 GENERAL REQUIREMENTS OF SAMPLING

J-1.1 The sampling instrument shall be clean and dry.

J-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

J-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both by suitable means or by rolling.

J-1.4 The samples shall be placed in suitable, clean, dry and airtight, metal, or dark or amber glass containers on which the material has no action.

J-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

J-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

J-1.7 Samples shall be stored in the dark.

J-2 SAMPLING INSTRUMENT

J-2.1 The following sampling instrument may be used:

- a) Sampling bottle or can, for taking samples from tanks or drums; and
- b) Sampling tube, for taking samples from bottles or small containers.

J-2.1.1 *Sampling bottle or can*, consists of a weighed glass or metal containers with removable stopper or top to which is attached a light chain (*see* Fig. 8). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

J-2.1.2 *Sampling tube*, made of metal or thick glass, 20 mm to 40 mm in diameter and 400 mm to 800 mm in length (*see* Fig. 9). The upper and lower ends are conical and reach 5 mm to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

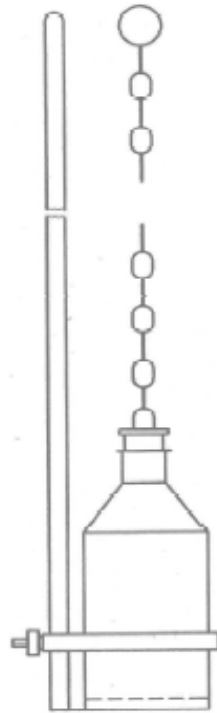
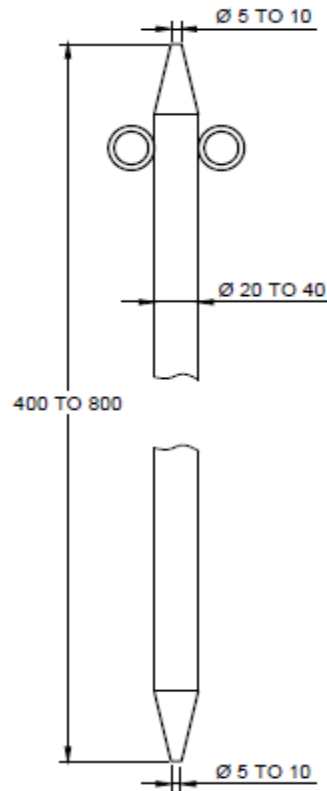


FIG. 8 SAMPLING BOTTLE



All dimensions in millimeters

FIG. 9 SAMPLING TUBE

J-2.1.2.1 For small containers the size of the sampling tube may be altered suitably.

J-3 SCALE OF SAMPLING

J-3.1 For Tanks and Drums, each tank or drum shall be sampled separately.

J-3.2 For Bottles and Small Containers, each lot (see J-3.2.1) shall be sampled separately.

J-3.2.1 Lot

In any consignment, all the containers of the same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different grades, the containers belonging to the same batch and grade shall be grouped together and each group shall constitute a separate lot.

J-3.2.2 The number of containers (n) to be selected from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

J-3.2.3 These containers shall be selected from the lot at random. In order to ensure the randomness of selection, a random number table may be used. For guidance and use of random number table, IS 4905 may be referred. In the absence of random number table, the following procedure may be adopted:

Starting from any container in the lot, count the containers as 1, 2, 3,, etc, up to r so on, in one order. Every r^{th} container thus counted shall be withdrawn to constitute the sample where r is the integral part of N/n (N and n being the lot size and sample size respectively).

J-4 TEST SAMPLE AND REFEREE SAMPLE

J-4.1 From Tanks and Drums

As far as possible, samples from a tank or drum should be drawn during the operation of filling. In

that case, equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material to ensure a fair amount of homogeneity. The total amount of the material collected shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

J-4.2 From Bottles and Small Containers

From each of the bottles or containers selected according to J-3.2.3, a small representative portion of the material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 500 ml. This shall be divided into three equal parts, one for the supplier and the third for the referee.

J-4.3 All the test samples shall be transferred to separate sample containers and sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

J-5 TESTS

J-5.1 Tests for the determination of all the requirements given in this specification shall be performed on the composite sample obtained as in J-4.1 or J-4.2.

J-6 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to this specification if all the test results satisfy the prescribed requirements.

Table 2 Scale of Sampling
(Clause J-3.2.2)

Sl No.	Lot Size	No. of Containers to be Selected
(1)	N (2)	n (3)
i)	Up to 15	3
ii)	16 to 40	4
iii)	41 to 65	5
iv)	66 to 110	7
v)	111 and above	10

ANNEX K

(Foreword)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

<i>Organization</i>	<i>Representative(s)</i>
National Chemical Laboratory (NCL), Pune	DR C. V. RODE (Chairperson)
Alkyl Amines Chemicals Ltd, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE (<i>Alternate</i>)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (<i>Alternate</i>)
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Ltd, Mumbai	SHRI KIRAN BHAT SHRI HEMAL (<i>Alternate</i>)
Chemical And Petrochemicals Manufacturers Association (CPMA), New Delhi	SHRI UDAY CHAND
CSIR — Central Drug Research Institute (CDRI), Lucknow	DR SANJEEV KANOJIYA
Deepak Fertilizer, New Delhi	DR L.B. YADAWA SHRI SURESH AMLE (<i>Alternate</i>)
Deepak Phenolics Ltd, Vadodara	SHRI DHARMESH SIDDHAPURIA SHRI SANDIP KUMAR PANDYA (<i>Alternate</i>)
Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizers, New Delhi	SHRI O. P. SHARMA SHRI VARUN SINGH POONIA (<i>Alternate</i>)
Dow Chemical International Pvt Ltd, Mumbai	SHRI V. MOHANDOSS SHRI GOVIND GUPTA (<i>Alternate</i>)
Godavari Biorefineries, Mumbai	SHRI SHANUL LAXMANRAO PAGAR SHRI APPASAHEB J. WANI (<i>Alternate</i>)
Gujarat Narmada Valley Fertilizers Company Ltd, Ahmedabad	SHRI R. M. PATEL SHRI C. S. PATEL (<i>Alternate</i>)
Hindustan Organic Chemicals Ltd (HOCL), Mumbai	SHRI DELEEP KUMAR K. SHRI PRADEESH NARAYANAN (<i>Alternate</i>)
India Glycols Ltd, Uttarakhand	DR R. K. SHARMA SHRI ALOK SINGHAL (<i>Alternate</i>)
Indian Chemical Council (ICC), Mumbai	DR MRITUNJAY CHAUBEY SHRI J. SEVAK (<i>Alternate</i>)
Indian Oil Corporation Ltd, Panipat	DR Y. S. JHALA
INEOS Styrolution India Ltd, Vadodara	DR KANAK DASS

<i>Organization</i>	<i>Representative(s)</i>
Jubilant Life Sciences Ltd, Noida	SHRI MANSUKH D. KANZARIYA
Laxmi Organic Industries, Mumbai	SHRI J. P. SURYAVANSHI DR VIJAY S. MISHRA (<i>Alternate</i>)
National Chemical Laboratory (NCL), Pune	DR RAVINDAR KONTHAM DR UDAYA KIRAN MARELLI (<i>Alternate</i>)
National Test House (NTH) , Kolkata	SHRI DEBASHIS SAHA DR GOPAL KRISHAN (<i>Alternate</i>)
Reliance Industrial Ltd (RIL), Mumbai	SHRI SREERAMACHANDRAN KARTHA SHRI VASANT WARKE (<i>Alternate</i>)
United Phosphorus Ltd (UPL), Mumbai	SHRI M. D. VACHHANI
In Personal Capacity (37 Nandanvan Society, Near GNFC Township, Narmadanagar, Bharuch – 392015)	DR MAYUR J. KAPADIA
BIS Director General	SHRIMATI MEENAL PASSI, SCIENTIST ‘F’/SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
KUMARI ADITI CHOUDHARY
SCIENTIST ‘B’/ASSISTANT DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

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